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(54) Pulverization of cellulosic material

(57) To facilitate cellulose powder production cellulose is embrittled with a difunctional compound eg 1,3 Bis(hydroxymethyl) imidazolidone and a wetting agent, dried, cured at a high temperature and then crushed without exposure to an acidic or alkaline washing treatments; the crushed cellulose may then be stripped free of the resin by a mild acid treatment to yield a cellulose powder without lowering the degree of polymerization of the cellulosic fibre.

SPECIFICATION

Pulverization of cellulosic materials

- 5 This invention relates to a new method for pulverization of Cellulosic Materials using Difunctional Agents

Methods for reducing the size of cellulosic materials generally fall into two categories, namely:

- 10 1 Chemical digestion methods which hydrolyse or otherwise degrade the fibre structure and lower the degree of polymerization.

- 2 Mechanical non-degradative methods which are quite involved and may not be efficient in reducing the size sufficiently and quantitatively.

- 15 Herein is described a method to reduce cellulosic materials in fibre, yarn or sheet form to micro-form without significantly lowering the Degree of Polymerization or substantially altering the main physical properties of the fibre.

- 20 If the cellulosic material is made sufficiently brittle by application of an appropriate cross-linking agent as used in Textile Resin Finishing, but at add-ons substantially greater, the resulting product would be so brittle that it can be crushed easily into micro-form powder.

- The same material would not behave in that manner, however, if after application of the resin, and prior to crushing, it was given an afterwash in an alkaline detergent bath.

- 30 The crushing stage can optionally be preceded by shredding of the modified material to any desired dimensions. The crushing process can be carried to the desired extent required by the end-use of the pulverized material.

- 35 The modified pulverized material can be used as such or, if the micro-form cellulose is required as the original unmodified state, it can be stripped free of the resin part by established methods using hot dilute mineral acids.

- 40 Alternatively the product can be washed in an alkaline detergent bath to remove the loosely adhering chemicals and partially restore the expected flexural properties of the fibre.

- 45 A specific embodiment of the invention will now be described by way of example with reference to a cotton fabric. The cellulosic material is soaked in a bath of:

- 1 Not less than 20%, on weight of fabric, of a difunctional crosslinking agent. The one used here is 1,3-Bis(hydroxymethyl)imidazolidone more commonly known by its abbreviation DMEU

- 2 15% $MgCl_2 \cdot 6H_2O$ on weight of resin

- 3 0.5-1.0% Wetting agent for example Sandozin

- 55 NI

Liquor-to-material ratio = 10:1 for a few minutes (say 5-10). Then the fabric is padded through a uniform pad mangle to a wet pick up of 90-100%.

- 60 The material is then dried at $\approx 80^\circ C$ for 10-15 minutes. The dry material is then cured at $160^\circ C$ for 3 minutes and kept dry till completely crushed.

- The whole material or, better, the shredded mass of material is crushed in a suitable crushing machine or by rubbing on a metal sieve or similar

rough surface. The crushed mass can then be sieved to separate appropriate parts as required.

- 70 The cotton fabric so treated as described above and crushed by rubbing manually on a metal sieve for a short period gave on sieving through a sieve of $45\mu m$ mesh a micro powder of dimensions less than $200\mu m$.

- 75 The natural cotton fibre has a diameter-to-length ratio of 1:1000. The crushing has reduced this ratio to less than 1:10. Ratios higher or lower than this can be achieved by arresting the crushing at the right extent.

- 80 The chemical digestive methods disrupt the structural integrity of fibres and the crystalline/amorphous order. They normally lower the degree of polymerization drastically. The mechanical non-degradative methods are expected to be cumbersome, costly and less efficient than the method proposed under this invention.

- 85 This method quantitatively and rapidly converts the cellulosic materials into micro-form without destroying the inherent physical properties or lowering the degree of polymerization of the cellulosic fibre.

- 90 All operations that require fibrous cellulose in microform may benefit from this method. Recycling of manufactured cellulosic articles for example rags, sheets and waste fibrous residues, is also envisaged to make good use of this method.

- 95 CLAIMS

1. A method of pulverization of cellulosic materials wherein the fibre, yarn or fabric is pulverized into powder without significantly altering the degree of polymerization of cellulose or affecting most of the other related physical properties of the fibre, such pulverization being due to embrittlement of the fibre through application of difunctional cross-linking agents as but at substantially higher add-ons than in Textile Resin finishing, drying, curing and crushing before any wetting or washing.

2. A method of pulverization of cellulose as claimed in Claim 1 wherein the cross-linking is effected by 21% of 1,3-Bis(hydroxymethyl)imidazolidone.

3. A method of pulverization of cellulose as claimed in Claim 1 or Claim 2 wherein the dimensions of the powder are controlled by the extent of crushing.

4. A method of pulverization of cellulose as claimed in any preceeding claim wherein the cross-linker is removed from the powder by a mild acid treatment to restore the normal physical properties of cellulose.

5. A method of pulverization of cellulose as claimed in any preceeding claim and substantially as described herein with reference to the description of the invention.